

## Corrosion Inhibition of Carbon Steel in 1M HCl Solution by 2-amino-1-methylbenzene (2-methylaniline)

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The inhibition effect of 2-amino-1-methylbenzene (AMB) on the corrosion of carbon steel in 0.5 M H<sub>2</sub>SO<sub>4</sub> was studied by weight loss, electrochemical impedance spectroscopy (EIS) techniques and potentiodynamic polarization methods. The results showed that AMB was a good inhibitor in 0.5 M H<sub>2</sub>SO<sub>4</sub> and inhibition efficiency increases with AMB concentration to attain 86.11% at 10<sup>-3</sup>M at 298 K. E(%) values obtained from various methods used are reasonably good agreement. The adsorption of AMB obeyed the Langmuir adsorption isotherm. Polarization curves showed that AMB acted as a mixed-type inhibitor in sulfuric acid. The effect of the temperature on the corrosion behavior with addition of 10<sup>-3</sup>M of the inhibitor was studied in the temperature range 298-328 K, and the thermodynamic parameters were determined and discussed.

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**Keywords:** Carbon Steel, EIS, Polarization, Weight loss, Acid inhibition, adsorption isotherm

### 1. INTRODUCTION

Corrosion can also be defined as a chemical reaction taking place at the surface of metals, which can be converted into a chemical compounds [1] Carbon steels are the most frequently used alloys due to the high ductility, which allows it for easier machining and welding [2]. Corrosion of metals/alloys is a natural phenomenon that can be controlled upon introducing small quantities of inhibitors into the corrosion environment. Several organic substances were studied for corrosion

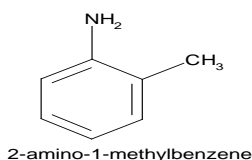
inhibition of metals/alloys at different corrosive environments [3-12]. Inhibitors are used in these processes to control metal dissolution as well as acid consumption. The uses of inhibitors are one of the most practical methods to inhibit corrosion of metals in many environments, especially in acidic media [13]. The role of inhibitors added in low concentrations to corrosive media is to decrease the dissolution of the metal with corrosive medium and is to inhibit the adsorption or coordination onto the metal surfaces [14, 15]. In the present work, the inhibition effect of 2-amino-1-methylbenzene (AMB) on corrosion of carbon steel in 0.5M H<sub>2</sub>SO<sub>4</sub> solution was studied using standard weight loss, potentiodynamic polarization curves and electrochemical impedance spectroscopy (EIS) methods. The adsorption isotherm of inhibitor on steel surface and the standard adsorption free energy ( $\Delta G_{ads}$ ) are obtained. The effect of temperature is studied and the thermodynamic parameters are also calculated and discussed in detail.

## 2. MATERIALS AND METHODS

### 2.1 Materials and inhibitor

The steel used in this study was a carbon steel (Euronorm: C35E carbon steel and US specification: SAE 1035) of chemical composition (wt%) of 0.370 % C, 0.230 % Si, 0.680 % Mn, 0.016 % S, 0.077 % Cr, 0.011 % Ti, 0.059 % Ni, 0.009 % Co, 0.160 % Cu, and the remainder iron (Fe). Coupons were cut into  $2 \times 2 \times 0.08$  cm<sup>3</sup> dimensions are used for weight loss measurements. Before the experiments the C38 steel samples were pre-treated by grinding with emery paper SiC (320, 800, and 1200 grade), rinsing with distilled water, degreasing by immersion in acetone in an ultrasonic bath for 5 min, washing again with bidistilled water, and then drying at room temperature. The acid solutions (0.5 M H<sub>2</sub>SO<sub>4</sub>) were prepared by dilution of analytical reagent-grade 98 % H<sub>2</sub>SO<sub>4</sub> with double-distilled water. The range of concentration of AMB was 10<sup>-6</sup> M to 10<sup>-3</sup> M.

The tested inhibitor, namely, Ammonium Iron was obtained 2-amino-1-methylbenzene (AMB) from Sigma–Aldrich chemical co. and his chemical structure is presented in Fig. 1.



**Figure 1.** shows the molecular structures of AMB

### 2.2. Measurements

#### 2.2.1. Weight loss measurements

Gravimetric measurements were performed after 6 h at room temperature, by use of an analytical balance (precision  $\pm 0.1$  mg). The carbon steel C38 specimens used were rectangular in

shape (length = 1.6 cm, width = 1.6 cm, thickness = 0.07 cm), and abraded by a series of emery paper (grade 320-800-1200) and then washed with distilled water, degreased with acetone, and finally dried at room temperature. The temperature was controlled at  $25 \pm 0.1^\circ\text{C}$  using a water thermostat. Gravimetric experiments were performed in a double glass cell equipped with a thermostated cooling condenser containing 80 mL non-de-aerated test solution. After immersion, the carbon steel specimens were withdrawn, carefully rinsed with bidistilled water, cleaning ultrasonically in acetone, dried at room temperature, and then weighed. Triplicate experiments were performed in each case and the mean value of the weight loss was calculated.

### 2.2.2. Electrochemical measurements

Electrochemical experiments were conducted by use of impedance equipment (Galvanostat/Potentiostat PGZ 100) controlled with Tacussel VoltaMaster 4 corrosion analysis software. A conventional three-electrode cylindrical Pyrex glass cell was used. The temperature was thermostatically controlled. The working electrode was C38 steel with the surface area of 1 cm<sup>2</sup>. A saturated calomel electrode (SCE) was used as reference. The counter electrode was a platinum plate of surface area 1 cm<sup>2</sup>. For polarization curves, the working electrode was immersed in test solution for 30 min or until a steady-state open circuit potential ( $E_{ocp}$ ) was obtained. The polarization curve was recorded by polarization from -800 to -200 mV/SCE with a scan rate of  $1\text{mV}\cdot\text{s}^{-1}$ . AC impedance measurements were performed in the frequency range 100 kHz – 10 mHz, with 10 points per decade, at the rest potential, after immersion in acid for 30 min, by applying 10 mV ac voltage peak-to-peak. Nyquist plots were obtained from these experiments. To produce the Nyquist plot, the data points were fitted to the best semicircle, by use of a non-linear least square fit, to give the intersections with the x-axis.

## 3. RESULTS AND DISCUSSION

### 3.1. Weight loss measurements

The corrosion rate ( $W_{corr}$ ) of carbon steel coupons in 0.5M  $\text{H}_2\text{SO}_4$  solution at different concentrations of AMB was determined after 6 h of immersion time at 298 K and calculated using the following equation:

$$W_{corr} = \frac{m}{S \cdot t} \quad (1)$$

Where  $m$  is the weight loss of C-steel sheets,  $S$  the total area of one C-steel sheets, and  $t$  is immersion time.

The obtained values of the gravimetric corrosion rates ( $W_{corr}$ ) and the inhibition efficiency ( $E_w$ ) are represented in Table 1. The inhibition efficiency ( $E_w$ ) (%) corrosion in the case of this method was calculated from the following equation:

$$E_w (\%) = \frac{W_{\text{corr}} - W'_{\text{corr}}}{W_{\text{corr}}} \times 100 \quad (2)$$

Where  $W_{\text{corr}}$  and  $W'_{\text{corr}}$  are the corrosion rates of carbon steel due to the dissolution in 0.5 M  $\text{H}_2\text{SO}_4$  in the absence and the presence of definite concentrations of AMB, respectively. The results obtained from gravimetric measurements show for inhibitor tested that the corrosion rate values decrease when the concentration of AMB increases. The analysis of Table 1 show that protection efficiency degree of surface coverage increased with with increasing concentration of inhibitor studied. This showed that AMB in solution inhibited the corrosion of carbon steel in 0.5 M  $\text{H}_2\text{SO}_4$ . The best action of this inhibitor is attained in the presence of  $10^{-3}\text{M}$  of AMB.

**Table 1.** Calculated values of corrosion rate ( $W_{\text{corr}}$ ), inhibition efficiency ( $E_w$  %) and degree of surface coverage ( $\theta$ ) for AMB in 0.5 M  $\text{H}_2\text{SO}_4$  corrosion of Carbon Steel from weight loss data.

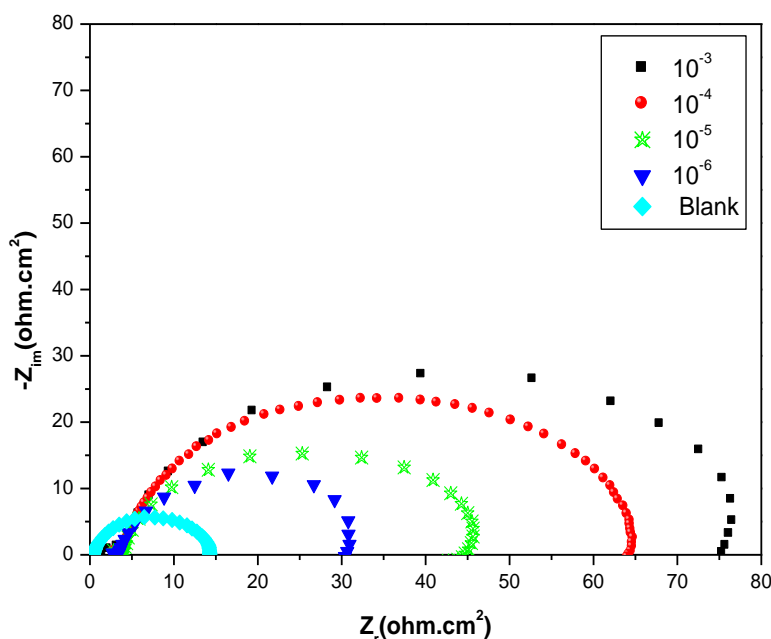
Inhibitor	Conc (M /L)	$W_{\text{corr}}$ ( $\text{mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$ )	$E_w$ (%)	$\theta$
Blanc	0	1.5482	-	-
AMB	$10^{-3}$	0.2322	85.00	0.8500
	$10^{-4}$	0.2861	81.52	0.8152
	$10^{-5}$	0.3867	75.02	0.7502
	$10^{-6}$	0.4718	69.52	0.6952

From the results, it was obvious that mild steel corroded in  $\text{H}_2\text{SO}_4$  solution. This is expressed in terms of weight loss of carbon steel. Upon addition of inhibitor (AMB), it was observed that the weight loss decreased as the corrosion rate and degree of surface coverage ( $\theta$ ). However, the weight loss decreased more as the concentration of inhibitor increased.

### 3.2. Electrochemical impedance spectroscopy (EIS).

The corrosion behavior of carbon steel in 0.5 M  $\text{H}_2\text{SO}_4$  solution in the absence and presence of different concentrations of the inhibitor was investigated by the EIS method at 298K after 30 min of immersion. Fig.3 show the Nyquist plot for carbon steel in 0.5 M  $\text{H}_2\text{SO}_4$  solution in the absence and presence of different concentrations of investigated inhibitor. The impedance diagrams have an approximately semi-circular appearance shows that the corrosion of carbon steel in 0.5 M  $\text{H}_2\text{SO}_4$  is controlled by a charge transfer resistance process. The capacitive loop at high frequency indicates that the corrosion of carbon steel is mainly controlled by a charge transfer process, as is usual in EIS studies, the high frequencies capacitive loop is related to the charge-transfer process occurring during metal corrosion and the double-layer behaviour [16]. The diameters of capacitive loops increase with the increase in AMB concentration, which indicates the increase of charge transfer resistance and improvement in inhibiting effect on carbon steel corrosion (Fig. 2). It is also observed that the shapes

of the impedance plots for the inhibited electrodes are not essentially different from those of the uninhibited electrodes.



**Figure 2.** Nyquist plots of the corrosion of C-steel in 0.5 M H<sub>2</sub>SO<sub>4</sub> without and with different concentrations of AMB at 298K.

The charge-transfer resistance values ( $R_{ct}$ ) were calculated from the difference in real impedance at lower and higher frequencies as suggested by Tsuru et al. [17]. To obtain the double-layer capacitance ( $C_{dl}$ ), the frequency at which the imaginary component of the impedance is maximum ( $-Z_{max}$ ) is found and  $C_{dl}$  values were obtained from the Eq. (3):

$$f(-Z_{max}) = \frac{1}{2 \cdot \pi \cdot C_{dl} \cdot R_{ct}} \tag{3}$$

With  $C_{dl}$ : Double layer capacitance ( $\mu\text{F} \cdot \text{cm}^{-2}$ );  $f_{max}$ : maximum frequency (Hz) and  $R_{ct}$ : Charge transfer resistance ( $\Omega \cdot \text{cm}^{-2}$ ).

The inhibition efficiency is calculated using charge-transfer resistance from Eq. (4):

$$E_{R_{ct}} (\%) = \frac{(R_{ct} - R_{ct}^0)}{R_{ct}} \times 100 \tag{4}$$

Where  $R_{ct}$  and  $R_{ct}^0$  are the charge transfer resistances in inhibited and uninhibited solutions respectively.

**Table 2.** Impedance parameters of C-steel in 0.5M H<sub>2</sub>SO<sub>4</sub> containing different concentrations of AMB.

Inhibitor	C (M/L)	R <sub>t</sub> (Ω.cm <sup>2</sup> )	f <sub>max</sub> (Hz)	C <sub>dl</sub> (μF/cm <sup>2</sup> )	E <sub>RT</sub> (%)
0.5M H <sub>2</sub> SO <sub>4</sub>	0	10	158	100.782	-
AMB	10 <sup>-3</sup>	72	60	36.86	86.11
	10 <sup>-4</sup>	60	75	35.38	83.33
	10 <sup>-5</sup>	40	125	31.85	75.00
	10 <sup>-6</sup>	32	158	31.49	68.75

Table 2 shows the EIS data where the C<sub>dl</sub> values decrease and the R<sub>ct</sub> values increase with the increase of the inhibitor concentrations. This is due to the gradual replacement of water molecules by the adsorption of the inhibitor molecules on the metal surface, and decreasing the extent of dissolution reaction. The high (R<sub>ct</sub>) values are generally associated with slower corroding system [18, 19]. The decrease in the C<sub>dl</sub> can result from the decrease of the local dielectric constant and/or from the increase of thickness of the electrical double layer [20] suggested that the inhibitor molecules function by adsorption at the metal/solution interface. [21, 22]. These results again confirm that AMB exhibit good inhibitive performance for C-steel in H<sub>2</sub>SO<sub>4</sub> solution. Inhibition efficiencies obtained from weight loss (E<sub>w</sub>), potentiodynamic polarization curves (E<sub>p</sub>) and EIS (E<sub>R<sub>ct</sub></sub>) are in good reasonably agreement.

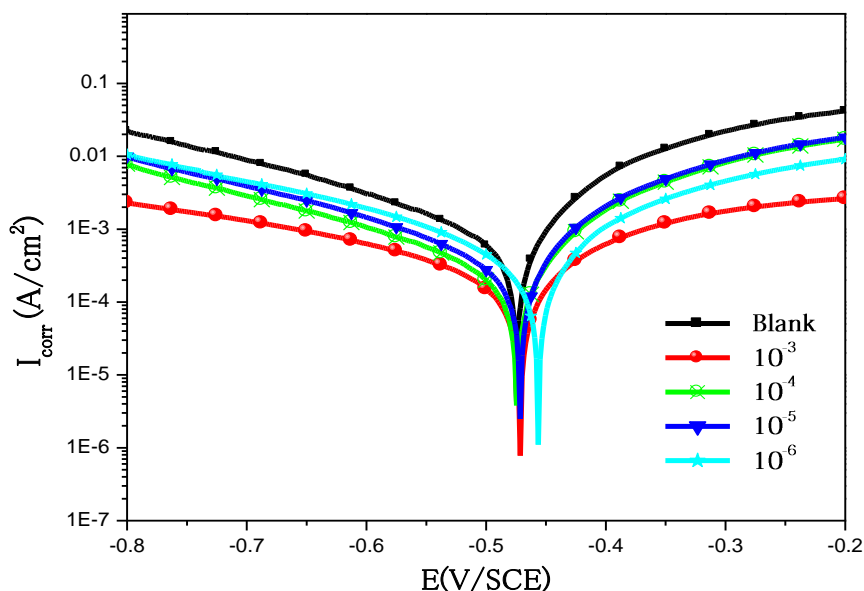
### 3.3. Potentiodynamic polarization curves

The polarization curves of carbon steel in acid solutions obtained with and without various concentrations of used inhibitor are shown in Fig.3. Electrochemical parameters such as corrosion current density (I<sub>corr</sub>), corrosion potential (E<sub>corr</sub>), constants cathodic Tafel slope (-β<sub>c</sub>), and the I<sub>corr</sub> values were used to calculate the inhibition efficiency, E<sub>p</sub> (%), by use of the equation:

$$E_I \% = \frac{(I_{corr}^o - I_{corr})}{I_{corr}^o} \times 100 \quad (5)$$

Where I<sub>corr</sub><sup>o</sup> and I<sub>corr</sub> are the corrosion current densities for steel electrode in the uninhibited and inhibited solutions respectively, determined by extrapolation of cathodic Tafel lines to the corrosion potential.

The electrochemical corrosion parameters including corrosion current densities (I<sub>corr</sub>), corrosion potential (E<sub>corr</sub>), cathodic Tafel slope (β<sub>c</sub>) and corresponding inhibition efficiency E<sub>I</sub> (%) are given in Table 3.



**Figure 3.** Polarization curves of C38 steel in 0.5M H<sub>2</sub>SO<sub>4</sub> containing different concentrations of AMB.

**Table 3.** Polarization data of C38 steel in 0.5M H<sub>2</sub>SO<sub>4</sub> without and with addition of inhibitor at 298 K.

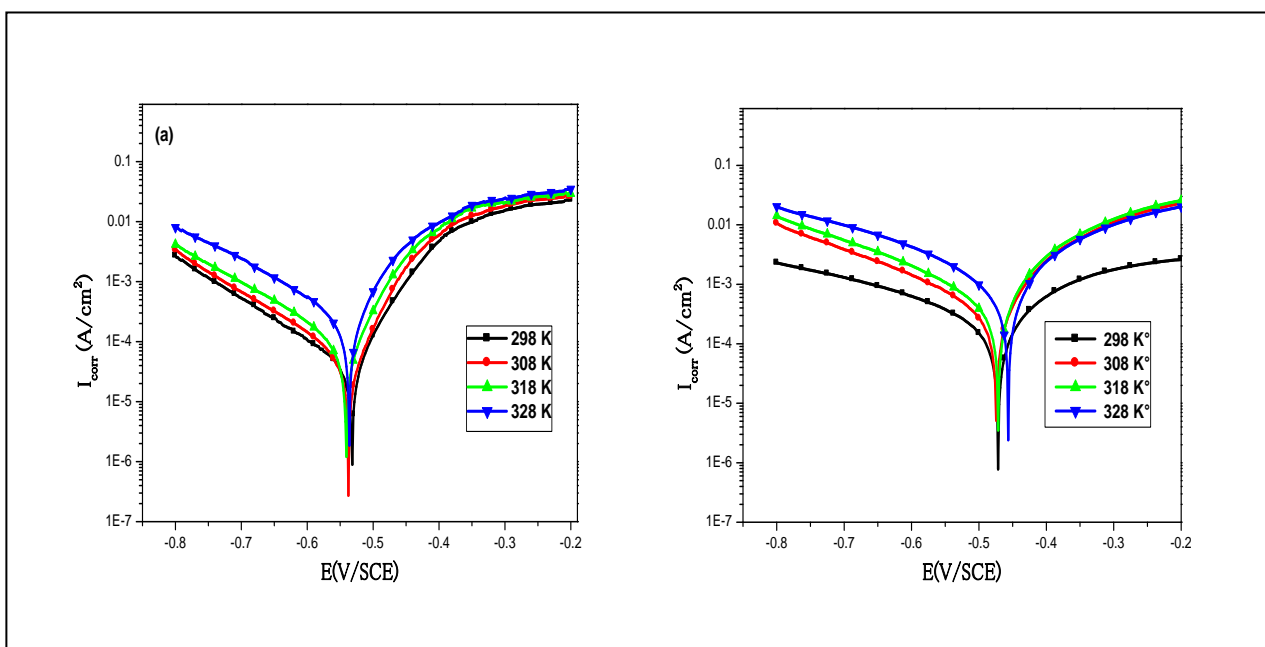
Inhibitor	Conc. (M)	E <sub>corr</sub> (mV/SCE)	I <sub>corr</sub> (μA/cm <sup>2</sup> )	-b <sub>c</sub> (mV/dec)	Ep (%)
Blank	0	478	1860	189	-
AMB	10 <sup>-3</sup>	471	263	258	85.86
	10 <sup>-4</sup>	475	320	231	82.79
	10 <sup>-5</sup>	470	445	184	76.07
	10 <sup>-6</sup>	456	551	246	70.38

The results showed in this Table indicated that the corrosion current density (I<sub>corr</sub>) decreases in the presence of inhibitors compared to the blank solution and also with increasing the inhibitor concentration which suggest that the presence of this compound retard the dissolution of steel in 1M HCl solution [23]. Presence of the inhibitor cause decrease in the corrosion rate, so shift both the anodic and cathodic curves to lower values of current densities. this result suggests that the addition of the inhibitor reduces the anodic oxidation of carbon steel and also retards the hydrogen reduction reaction. This reduction of the corrosion process can be attributed to the covering of adsorbed inhibitor molecules on the steel surface. [24]. The slope of the cathodic Tafel lines (β<sub>c</sub>) are slightly changed on increasing the concentration of the investigated compound. This means that there is no change of the mechanism of the inhibition in presence and absence of inhibitors and that inhibitors affects both anodic and cathodic reactions, so it is mixed type inhibitors. Inspection of polarization curves and electrochemical parameter (table.3) reveals that cathodic current densities decrease with the increase of

the inhibitor concentration and the inhibition efficiency  $E_p$  (%) increases with the inhibitor concentration up to a maximum of 85.86 %, due to the increase in the blocked fraction of the electrode surface by adsorption.

### 3.4. Effect of temperature.

To assess the influence of temperature on corrosion and corrosion inhibition processes, polarization tests were carried out at various temperatures (25–55°C) in the absence and presence of  $10^{-3}$  M of AMB, as shown in Fig. 4.



**Figure 4.** Polarization curves of mild steel in 0.5 M  $H_2SO_4$  solution in the absence (a) and presence (b) of  $10^{-3}$  M of AMB at different temperatures after 30 min immersion

**Table 4.** Electrochemical parameters of the corrosion of carbon steel in 0.5 M  $H_2SO_4$  solutions in the absence and presence of  $10^{-3}$  M of inhibitor.

Inhibitor	T(K)	$E_{corr}$ (mV/SCE)	$I_{corr}$ ( $\mu A/cm^2$ )	$-b_c$ (mV/dec)	E (%)
Blank	298	-478	1860	190	-
	308	-483	2754	151	-
	318	-493	3036	126	-
	328	-497	4511	104	-
AMB	298	471	263	258	85.86
	308	474	435	233	84.20
	318	472	535	226	82.38
	328	456	925	201	79.49



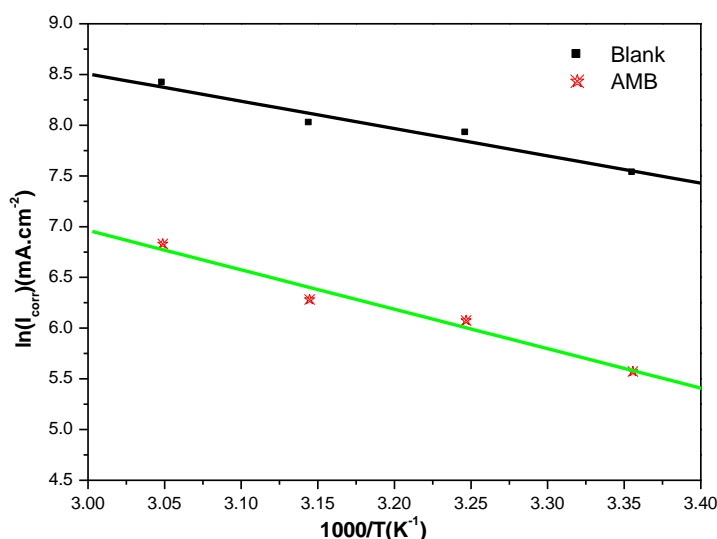
Corresponding electrochemical data are given in Table 4. As seen from Fig.4 and Table 4, the corrosion current density increases with increasing temperature, in both uninhibited and inhibited solutions. Inhibition efficiency for AMB decreases with increase in temperature and a slight changes in there values are observed in the range of temperature studied.

From this result, it can be concluded that the value of corrosion current density increases in the absence and the presence of inhibitor. After addition of the tested inhibitor in corrosive medium, the dissolution of carbon steel is extensively retarded.

To calculate the activation parameters of the corrosion process, Arrhenius equation. (6) was used. [25]

$$I_{corr} = A \exp\left(-\frac{E_a}{RT}\right) \quad (6)$$

Where k is the pre-exponential factor,  $E_a$  is the apparent activation energy of the corrosion process, R is the gas constant and T is the absolute temperature.



**Figure 5.** Arrhenius plots of carbon steel in 0.5M H<sub>2</sub>SO<sub>4</sub> with and without 10<sup>-3</sup>M of AMB.

Fig. 5 shows Arrhenius plots of the logarithm of the current density vs 1/T for carbon steel in the corrosive medium with and without addition of 10<sup>-3</sup> M of AMB. Straight lines are obtained with a slope of (-E<sub>a</sub>/R).

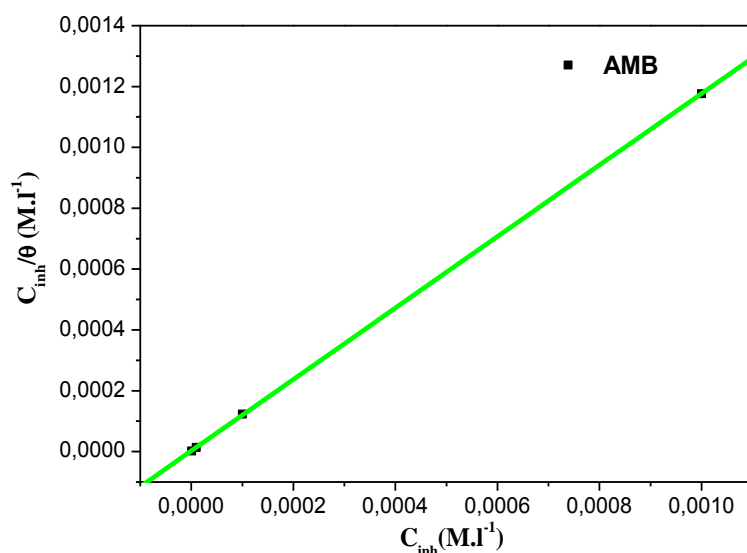
The value of  $E_a$  can be obtained from the slope of the straight line which was found to be 22.37 kJ .mol<sup>-1</sup> and 32.29 kJ mol<sup>-1</sup> in the absence and presence of 10<sup>-3</sup>M of AMB, respectively. The higher value of  $E_a$  in the presence of AMB than its absence indicates a strong inhibitive action of the AMB by increasing the energy barrier for the corrosion process.[26] And the higher  $E_a$  value in the inhibited solution can be correlated with the increased thickness of the double layer.

3.5. Adsorption isotherm

Adsorption isotherms are very important to understand the mechanism of inhibition corrosion reactions. The curve obtained clearly shows that the data fit well with Langmuir adsorption isotherm (Fig.6) was found to be the best description of the adsorption behavior of the studied inhibitor, which obeys [27]:

$$\frac{C}{\theta} = \frac{1}{K} + C \tag{8}$$

Where C is the concentration of inhibitor, K the adsorptive equilibrium constant, and  $\theta$  is the fraction of the surface covered calculated as follows  $\theta = E(\%)/100$ .



**Figure 6.** Langmuir isotherm adsorption mode of AMB on the carbon steel surface in 0.5M H<sub>2</sub>SO<sub>4</sub> at 298K from EIS.

Plots of C/θ against C yield straight lines as shown in Fig. 6, and the corresponding linear regression parameters are listed in Table 5. Both linear correlation coefficient (R) and slope are very close to 1, indicating the adsorption of AMB on carbon steel surface obeys the Langmuir adsorption isotherm in sulfuric acid solutions. The adsorptive equilibrium constant (K) is related to the standard free energy of adsorption (ΔG<sub>ads</sub>) as shown the following equation [27]:

$$K = \frac{1}{55.5} \exp\left(-\frac{\Delta G_{ads}}{RT}\right) \tag{10}$$

ΔG<sub>ads</sub> is the standard free energy of adsorption reaction, R is the universal gas constant, T is the absolute temperature (K) and the value of 55.5 is the concentration of water in the solution in mol/L.

**Table 5.** Parameters of the straight line of  $C/\theta$  against  $C$  and adsorption free energy  $\Delta G_{\text{ads}}$ .

Inhibitor	Slope	$K_{\text{ads}} (\text{M}^{-1})$	$R^2$	$\Delta G_{\text{ads}} (\text{kJ/mol})$
BNPP	1.17	441904.9639	0.99999	-42.136

Large values of  $K_{\text{ads}}$  mean good inhibition efficiency of the inhibitor and strong electrical interaction between the adsorbate and the adsorbent.

The negative values of  $\Delta G_{\text{ads}}$  indicate that the adsorption of inhibitor molecule on steel surface is spontaneous and also the strong interaction between inhibitor molecules and the metal surface [28, 30-31]. Generally, values of  $\Delta G_{\text{ads}}$  up to  $-20 \text{ kJ. mol}^{-1}$  are consistent with the electrostatic interaction between the charged molecules and the charged metal (physical adsorption), the inhibition acts due to the electrostatic interactions between the charged molecules and the charged metal, while the values around  $-40 \text{ kJ/mol}$  or smaller, were seen as chemisorption, which is due to the charge sharing or a transfer from the inhibitor molecules to the metal surface to form a covalent bond [32-33]. In the present work, the value of  $\Delta G_{\text{ads}}$  is found to be around  $-40 \text{ kJ.mol}^{-1}$ ; means that the adsorption mechanism of AMB on carbon steel surface is mainly the chemisorption.

#### 4. CONCLUSION

The investigated compound is a good inhibitor for the corrosion of Carbon steel in 1M HCl solutions and their act as mixed type inhibitor. The results obtained from all the electrochemical measurements show that the inhibition properties increase with small differences in their efficacy values. Double layer capacitances decrease with respect to blank solution when these compounds were added. This fact may explained by adsorption of these molecules on the Carbon steel surface. The adsorption of the investigated compound on carbon steel surface in HCl solution follow Langmuir adsorption isotherm. The negative values of  $\Delta G^{\circ}_{\text{ads}}$  show the spontaneity of the adsorption process. The parameter of adsorption free activation energy  $\Delta G^{\circ}_{\text{ads}}$  indicates that the adsorption of inhibitor involve chemisorption.

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